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CONTRACT NO: DAMD17-90-C-0078

TITLE: DEVELOPMENT OF NEW LASER PROTECTIVE DYES

PRINCIPAL INVESTIGATOR: Dennis P. Pacheco, Ph.D.

CONTRACTING ORGANIZATION: Spectra Science Corporation
300 Metro Center Blvd.
Warwick, Rhode Island 02886

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Warwick, Rhode Island 02886

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The overall purpose of this research effort was to develop dyes that are compatible with optical grade plastic that can be manufactured into lens devices, and will provide protection for the eye against lasers that emit in the region from 700 nm to 1100 nm.

In Phase II, a number of new laser-protective dyes were synthesized and tested in optical plastics. These dyes were drawn from five different chemical families: squarates, croconates, fluorenes, methines, and metal-complex dyes. Also, methods of theremally and photochemically stabilizing these dyes have been investigated and developed. The basic strategy of the program is shown schematically on page 3 of report. Sets of lenses from several dyes or dye systems have been produced and will be delivered to the contractor for evaluation.

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## SECTION I - INTRODUCTION

The overall purpose of this research effort was to develop dyes that:

are compatible with optical grade plastic that can be manufactured into lens devices and

will provide protection for the eye against lasers that emit in the region from 700 nm to 1100 nm.

One of the major problems with dyes available for this purpose is that they are thermally and/or photochemically unstable. In Spectra Science's Phase I research, the feasibility of thermally stabilizing cyanine and squarylium dyes for simulated polycarbonate injection–molding conditions was demonstrated, justifying the continuation of Spectra Science's dye synthesis and evaluation program. In Phase II, a number of new laser-protective dyes were synthesized and tested in optical plastics. These dyes were drawn from five different chemical families: squarates, croconates, fluorenes, methines, and metal-complex dyes. Also, methods of thermally and photochemically stabilizing these dyes have been investigated and developed. The basic strategy of the program is shown schematically on the following page. Sets of lenses from several dyes or dye systems have been produced and will be delivered to the contractor for evaluation.

## SECTION II - PROGRAM HIGHLIGHTS

### A. DYES SYNTHESIZED DURING THE PROGRAM

Table 1 lists the dyes that have been synthesized during this program with $\lambda_{max}$ greater than 700 nm. The structures of these dyes are shown in Figure 1. Because of the sheer number of these compounds, we will not attempt to discuss the synthetic routes here. The individual monthly reports can be consulted for details.

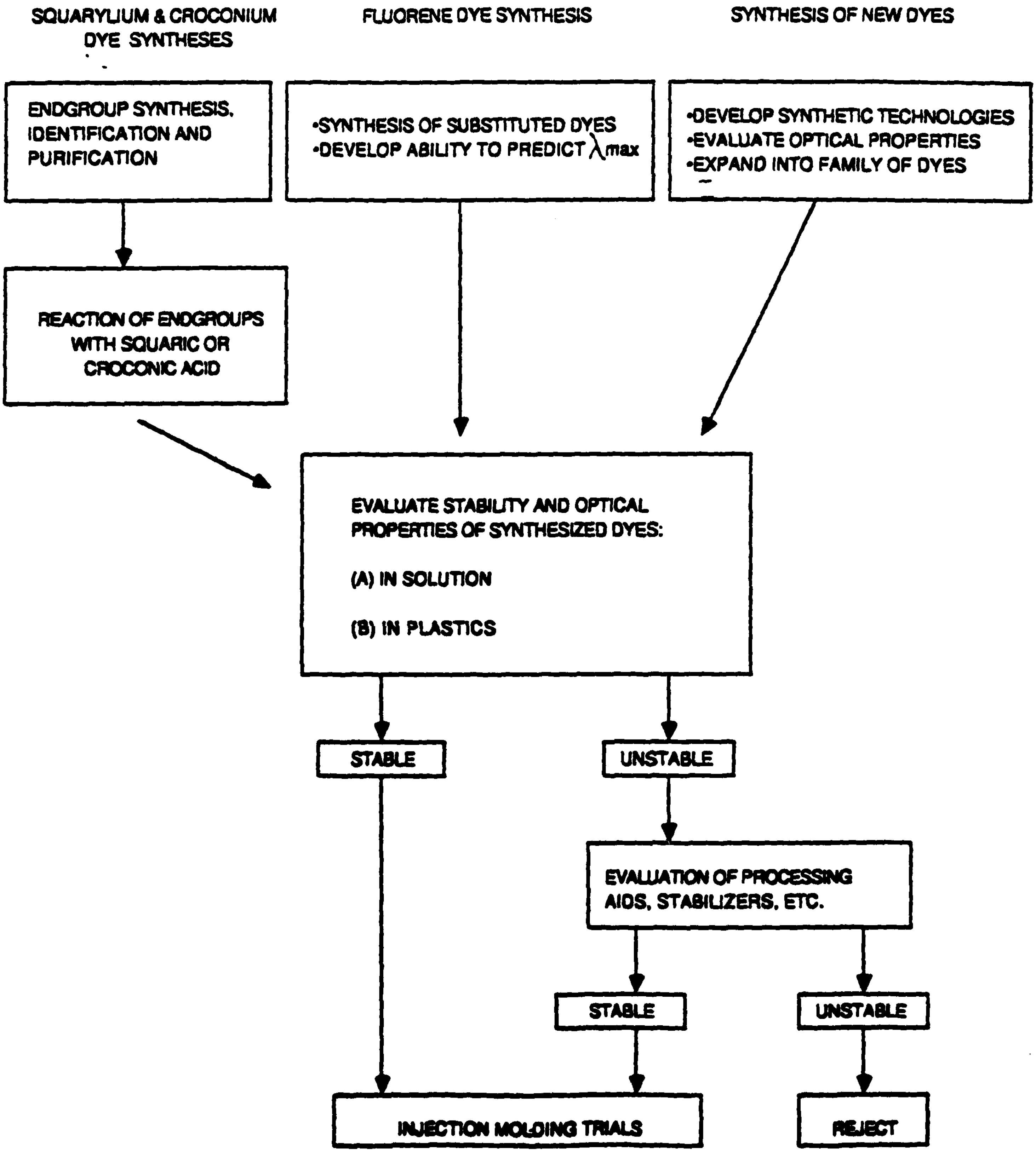

SQUARYLIUM & CROCONIUM DYE SYNTHESES
FLUORENE DYE SYNTHESIS
SYNTHESIS OF NEW DYES
ENDGROUP SYNTHESIS, IDENTIFICATION AND PURIFICATION
•SYNTHESIS OF SUBSTITUTED DYES
•DEVELOP ABILITY TO PREDICT λmax
•DEVELOP SYNTHETIC TECHNOLOGIES
•EVALUATE OPTICAL PROPERTIES
•EXPAND INTO FAMILY OF DYES
REACTION OF ENDGROUPS WITH SQUARIC OR CROCONIC ACID
EVALUATE STABILITY AND OPTICAL PROPERTIES OF SYNTHESIZED DYES:
(A) IN SOLUTION
(B) IN PLASTICS
STABLE
UNSTABLE
EVALUATION OF PROCESSING AIDS, STABILIZERS, ETC.
STABLE
UNSTABLE
INJECTION MOLDING TRIALS
REJECT

**Table 1**

**Dyes with Absorption Maxima ($\lambda_{max}$) Greater than 700 nm**
**Synthesized by Spectra Science During this Program**

| Dye | Dye Classification | $\lambda_{max}$ (nm)[1] |
|---|---|---|
| 1044 | Croconium | 823 |
| 1045 | Squarylium | 765 |
| 1049 | Squarylium | 1057 |
| 1096 | Squarylium | 742 |
| 1097 | Squarylium | 733 |
| 1098 | Squarylium | 912 (DMF) |
| 1099 | Croconium | 846 (DMSO) |
| 1100 | Squarylium | 988 (TECE) |
| 1101 | Squarylium | 905 (TECE) |
| 1106 | Squarylium | 711 |
| 1107 | Squarylium | 1150 (TECE) |
| 1109 | Squaramide | 835 (DMF) |
| 1112 | Squarylium | 1010/1115/1100 (TECE) |
| 1115 | Squarylium | 910 (TECE) |
| 1116 | Squarylium | 905/1075 |
| 1117 | Squarylium | 870/965/1140 |
| 1118 | Squarylium | 980 |
| 1119 | Squarylium | 606/650/852 (TECE) |
| 1120 | Squarylium | 725 (TECE) |
| 1122 | Squarylium | 1038 (TECE/MeOH) |
| 1123 | Squarylium | 952 (TECE/MeOH) |
| 1124 | Squarylium | 950 (TECE/MeOH) |
| 1125 | Squarylium | 950 (TECE) |
| 1126 | Squarylium | 1015 (TECE) |
| 1127 | Squarylium | 1000 |
| 1128 | Squarylium | 820 |
| 1129 | Squarylium | 1000 (TECE) |
| 1131 | Squarylium | 888 |
| 1137 | Squarylium | 970 (TECE) |
| 1138 | Squarylium | 1010 |

| | | |
|---|---|---|
| 1139 | Fluorene | 455/965 (MeOH) |
| 1140 | Fluorene | 455/965 (TECE) |
| 1141 | Fluorene | 725 (Ac) |
| 1145 | Fluorene | 500/1360 (Ac) |
| 1148 | Fluorene | 980 (Ac) |
| 1149 | Squarylium | 687 |
| 1150 | Squarylium | 880 |
| 1151 | Croconium | 850 |
| 1152 | Fluorene | 900, 1040 (Ac) |
| 1153 | Fluorene | 1000 (Ac) |
| 1155 | Squarylium | 980 (TECE) (LOW CONC.)<br>1060 (TECE) (HIGH CONC.) |
| 1156 | Squarylium | 960(TECE) |
| 1157 | Squarylium | 890 |
| 1159 | Squarylium | 980 (TECE) |
| 1160 | Squarylium | 605, 815 |
| 1161 | Methine | 335, 767 |
| 1166 | Squarylium | 770 |
| 1168 | Squarylium | 910 |
| 1169 | Ni Complex | 790 (DMSO) |
| 1170 | Cu Complex | 775 |
| 1171 | Ni Complex | 779 |
| 1172 | Cu Complex | 759 |
| 1173 | Ni Complex | 768 |
| 1175 | Squarylium | 668/796 |
| 1177 | Methine | 754 |
| 1178 | Methine | 728 |

FOOTNOTE:

$\lambda_{max}$ values obtained in methylene chloride unless otherwise specified:

- (TECE) = 1,1,2,2, tetrachloroethane
- (DMF) = dimethylformamide
- (MeOH) = methanol
- (DMSO) = dimethylsulfoxide
- (Ac) = acetone

## B. INCORPORATION OF DYES INTO PLASTIC

In this program, a number of candidate NIR laser-protective dyes were evaluated in plastic media. These dyes are listed in Table 2.

<u>Table 2</u>

**Dye/Plastic Combinations Investigated during this Program**

| DYE | PLASTIC* | λmax (nm) |
|---|---|---|
| SS-1044 | PETG | 834 |
| | PC | --- |
| SS-1045 | PC | 777 |
| SS-1049 | PETG | ≈ 1070 |
| | PC | |
| SS-1101 | PMMA | 850/990 |
| SS-1106 | PC | 717 |
| SS-1106/SS-1045 | PC | 700-800 |
| SS-1107 | PETG | 1070 |
| | PC | |
| SS-1116 | PETG | 905 |
| | PC | 905 |
| SS-1118 | PETG | 985 |
| | PC | 975 |
| SS-1145 | PETG | --- |
| SS-1150 | PETG | 893 |
| | PC | |
| SS-1151 | PC | 860 |
| SS-1155 | PETG | 1060 |
| | PC | |
| SS-1159 | PC | 980 |
| SS-1161 | PC | 755 |

* PETG-Polyethylene terphthalate glycolate, PC-Polycarbonate, PMMA-polymethyl methacrylate

## B-1 Testing Methodology

In the early part of this program, dyes were incorporated into plastic through solution casting. That is, the plastic and dye were co-dissolved in a suitable medium, such as methylene chloride. The resulting solution was then spread in a thin layer on a glass plate. The volatile solvent was allowed to evaporate, leaving a thin sheet of dye-impregnated plastic for testing. The obvious disadvantage of this approach is that commercial extrusion or injection-molding processes expose the dye to elevated temperatures and shear for a period of minutes, and this is absent from the solution casting.

The next stage of testing involved construction of a "hot-melt" apparatus, in which molten plastic and dye are thoroughly mixed with a time-temperature profile (200°-300° C for a period of 2-3 minutes) approximating that for commercial processing. This led to a more realistic assessment of the performance of our NIR dyes. Even this approach is idealized in that it does not include the shear (and the associated local heating problems) of extrusion or injection-molding. It does have the advantage of allowing real-time, in-house testing of new dyes. The compounded material was then pressed into thin wafers for spectral analysis.

Dyes that performed well in the hot-melt apparatus were then impregnated into plastic using an outside extruder or injection-molder. Time-temperature profiles in these machines were selected to closely match the conditions for producing commercial lens blanks. Sometimes special processing conditions were needed to produce good dye performance; these are described in the sections below for each dye.

## B-2 Dyes Developed for 1064 nm ($\lambda_3$)

Because of the importance of this wavelength for industrial, medical, and military applications, we will discuss $\lambda_3$ absorbers first. During this program, Spectra Science developed three dyes for 1064 nm: SS-1049, SS-1107, and SS-1155. The last of these was synthesized relatively late in the program, and so is less well studied. All are characterized by a neutral-gray appearance in lens blanks, which is an advantage over the conventional green $\lambda_3$ dye in some areas (e.g., color-balancing).

Early work on SS-1107 and SS-1049 yielded relatively low photopic values in PC (approximately 16-18% for an OD of four). A major breakthrough occurred when molecular oxygen was removed from both the plastic pellets and the atmosphere in the extruder/injection molder. The first time this was implemented, the photopic values jumped to the 25-27% level in PC (for an OD = 4). A detailed study of the effect of oxygen was presented in the October, 1992 Report. By being more scrupulous in the removal of oxygen and by minimizing the processing temperature of the PC, photopic values were further improved to about 35 - 37% (OD = 4).

The results from a series of extrusion tests performed on SS-1107 and SS-1049 in both PC and PETG are included in Table 3.

**Table 3**
**Photopic/Scotopic Transmission**
**Extrapolated to IR Absorbance = 4.0**

| Plastic | SS-1107 | | SS-1049 | |
|---|---|---|---|---|
| | Photopic | Scotopic | Photopic | Scotopic |
| PC | 33.2 | 39.1 | 24.5 | 22.8 |
| PETG | 40.6 | 44.9 | 40.2 | 39.0 |

The next step was to demonstrate that comparable performance could be achieved by a *commercial* injection–molder using conventional equipment. To this end, four lots of lenses were injection-molded with SS-1107 in PC at Bollé in France. The targeted absorbance was 6.0. Photopic/scotopic measurements made directly on the lenses showed numbers very similar to those achieved at Spectra Science.

A lens sample containing SS-1107 was forwarded to J. Lund of the U.S. Army for laser saturation tests. Subsequent measurements in his laboratory verified that this dye passes saturation requirements for laser eye protection. This result, together with the photopic/scotopic performance, demonstrates the suitability of this material for commercial production of polycarbonate lenses.

Finally, a number of lenses containing two and three Spectra Science dyes were fabricated at Bollé to characterize optical performance with multiple–line protection. Specifically, lenses were molded to protect against laser wavelengths: (a) $\lambda_3$, (b) $\lambda_3 + \lambda_2$, and (c) $\lambda_3 + \lambda_2 + \lambda_1$. The lenses were prepared by injection–molding vacuum–deoxygenated polycarbonate pellets that had been coated with the following dyes:

$\lambda_1$: SS-1037, $\lambda_2$: SS-1093, $\lambda_3$: SS-1107

The spectral and photopic properties of these experimental lenses are presented in Table 4.

**Table 4**
**Optical Performance of Lenses Containing Dyes and Dye Mixtures Capable of Protecting Against Various Laser Wavelengths**

| | TARGET ABSORBANCE | | | Measured |
|---|---|---|---|---|
| Lens | 1064 | 694 | 532 | Photopic Visual % transmission |
| $\lambda_3$ | 4 | — | — | 34.9 |
| $\lambda_3 + \lambda_2$ | 4 | 4 | — | 25.8 |
| $\lambda_3 + \lambda_2 + \lambda_1$ | 4 | 4 | 2 | 9.3 |

Samples of each of these dyes systems will be provided to the Army at the end of the program.

In the February, 1993 monthly, we reported the synthesis of a new squaric acid dye (SS-1155) absorbing at 1060 nm ($\lambda_3$) in TECE. Shortly thereafter, this compound was evaluated in both PC and

PETG. Results are given in Table 5. Table 6 shows the effects of additives on the spectra of three identical samples (40 mg SS-1155 / 1/2 lb PETG). Since we had seen significant changes in the spectral profile of liquid solutions when we added either of the starting materials (squaric acid or end groups) to the SS-1155 dye solution, we wanted to explore their effects in extruded plastic. While we did not observe spectral shifts of any note, the maximum absorbance changed as listed in Table 6. The results to date for this dye have not been as good as those for SS-1107, although it has not been studied as extensively due to its synthesis late in the program.

**Table 5**
**Extrusion Results for SS-1155 in Plastic**

| PLASTIC | $\lambda$max | PHOTOPIC | SCOTOPIC |
|---|---|---|---|
| Polycarbonate | 1064 | 29.0 | 27.1 |
| PETG | 1064 | 37.7 | 36.6 |

**Table 6**
**Effect of Additives on the Absorption of SS-1155 in PETG**

| ADDITIVE | AMOUNT | $\lambda$max | Imax |
|---|---|---|---|
| None | NA | 1065 | 0.67 |
| Squaric Acid | 40 mg | 1065 | 0.84 |
| Squaric Acid | 10 mg | 1065 | 0.84 |
| + Endgroup | 40 mg | | |

## B-3 Dyes to Cover the 700-800 nm Region (Alexandrite)

In order to determine mutual compatibility and broad-band absorption capabilities of Spectra Science NIR dyes, SS-1106 and SS-1045 were co-processed into GE High-Flow polycarbonate resin. Individually, these two dyes have peak absorptions ($\lambda$max) at 717 and 777 nm, respectively. These particular dyes were chosen because, together, they cover the tuning range of the Alexandrite laser (700 to 800 nm), which is finding increased use in both military and commercial applications and environments.

Initially, several dye-doped wafer samples were prepared using the thermoplastic polymer mixing apparatus described in the September 1992 report. The mixing conditions were approximately two and one half minutes at 215° to 225°C. These mixing conditions were chosen to simulate those encountered in the commercial injection-molding of PC lenses. The compounded SS-1106 and SS-1045 dye containing PC resin was then pressed into a 0.14 mm thick film by heating the resin between chrome plates (in a hot press) for approximately one and one half minutes at 190°C. The absorption spectrum of this wafer showed an optical density of 1.0-1.5 in the 700 to 800 nm wavelength range. This extrapolates to about 15 for a 2-mm thick lens. Clearly, the concentrations used in this experiment are much higher than that needed for a typical military laser-protective lens application (OD approximately 4 to 6).

After successful extrusion tests of this dye combination, we went on to the injection-molding of lens blanks. By this time, we had to arrange for another commercial vendor to injection-mold the pieces. (Our previous vendor, Bollé of France, was unavailable for trials.) The resulting lens blanks were of high quality with good dye performance (i.e., no evidence of dye degradation). The optical densities were considerably higher than anticipated, because the lenses are 3.1 mm thick, rather than the 2 mm we expected. The estimated absorbance for these pieces is 16.5 for the SS-1045 peak (776 nm), and 13.2 near the SS-1106 peak (734 nm). These values are extrapolated from thin wafers pressed from the thick lens blanks. (The absorption spectrum of one such wafer is included as Figure 2.)

Overall, Figure 2 represents a promising materials development. Noteworthy is the high optical transmission in the visible range. The ratio of peak absorbance in the IR to that for 550 nm is 25 to 30, indicating these photopic values should be very attractive for lenses utilizing these dyes. Furthermore, by adjusting the relative concentration of the two dye components, the absorption profile in the 700 to 800 nm range can be rendered quite flat.

## B-4 Dyes for 800-900 nm Absorption

In this spectral region, three dyes were successfully extruded into PETG and/or PC:

(a.) the croconium dye SS-1044 ($\lambda_{max}$ = 834 nm in PETG).

(b.) the squarylium dye SS-1151 ($\lambda_{max}$ = 860 nm in PC).

(c.) the squarylium dye SS-1150 ($\lambda_{max}$ = 893 nm in PETG).

(As discussed in Section B-2, deoxygenated resin was used.) The absorption spectra of SS-1150 in PETG and SS-1151 in PC are shown in Figures 3 and 4, respectively. Note in particular the high visible transmission of these samples. The strong peak at 893 nm for SS-1150 is very close to the emission of a semiconductor laser at approximately 900 nm. Similarly, the absorption peak for SS-1151 covers the 850 nm emission of another semiconductor device.

SS-1044 and SS-1150 were also tested in PC. SS-1150 performed very well, with a strong absorption peak at approximately 900 nm, and low visible absorption. (The spectrum is very similar to that shown in Figure 3.) The estimated photopic transmission for an OD = 4 is approximately 45%.

For SS-1044 in PC, on the other hand, the results were not so favorable. Apparently, much of the dye degraded during the extrusion process.

## B-5 Dyes for 900 - 1000 nm Absorption

For this spectral region, new dyes synthesized in this program include SS-1116 (905 nm in PC), SS-1118 (975 nm in PC), SS-1101 (with peaks at 850 and 990 nm in PMMA), and SS-1159 (980 nm in PC). Of these, SS-1101 and SS-1159 are particularly promising in that they show good visible transmission in PC and provide relatively broad coverage from about 850 - 1050 nm. Potential laser applications include protection against Ti:sapphire, which is tunable from 800 to 1000 nm.

The absorption spectrum of SS-1159 extruded into PC is shown in Figure 5. Deoxygenated resin was used to maximize photopic performance. The ratio of peak IR absorbance to absorbance at 550 nm is approximately 12 for this dye, which makes it an attractive candidate for this spectral region. In July, samples of both SS-1159 and SS-1101 were prepared for outside injection-molding trials. The results are not available at this time.

B-6 Dyes for Wavelengths beyond 1200 nm

Our best candidate for longer wavelengths in the NIR was the fluorene dye SS-1145. As discussed in the Sixth Quarterly Report, Spectra Science has synthesized a number of new fluorene compounds covering the 750-1400 nm region. These dyes proved difficult to make in high yield, and most have relatively low decomposition temperatures (<250 °C). One dye however, SS-1145, has a peak absorption at 1360 nm (in acetone) and a decomposition temperature >300 °C. This compound was prepared in sufficient quantity (60 mg) for extrusion tests using deoxygenated resin. The resulting material, however, showed little absorption anywhere in the NIR, indicating that little of the fluorene dye survived the extrusion process.

# SECTION III - ADMINISTRATIVE COMMENTS

During this Phase II SBIR project, Spectra Science succeeded in synthesizing over 50 new dyes that have a $\lambda_{max}$ between 700 and 1400 nm. Emphasis was placed on creating stable, "notch" absorbers having minimum optical activity in the visible. By using these dyes in combination, an extended spectral range can be covered.

In making these compounds, new synthetic routes had to be developed. In the vast majority of cases, the dyes can be made reproducibly and in commercially useful quantities (tens of grams or larger). This synthetic activity represented a major effort, as numerous problems were encountered and had to be solved.

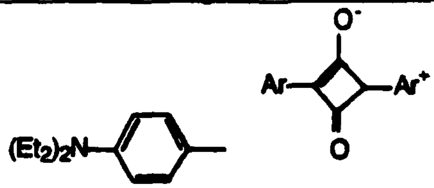
One of the dyes synthesized for 1064 nm, SS-1107, has been shown to be injection-moldable into PC with excellent transmission properties. (Photopic values of 35% and higher have been achieved at an OD = 4.) Because the absorption spectrum of this dye has a minimum near 480 nm, scotopic values are several percent higher, which is important for some military applications. Lens blanks produced using this dye have a neutral-gray appearance, which is an advantage over the conventional green lenses in many cases. SS-1107 has passed laser saturation tests performed by the Army.

A number of other dyes synthesized during this program have been molded or extruded into PC and/or PETG to cover specific wavelengths in the 700 -1100 nm range. In most cases, they can be used in various combinations to provide broad-band spectral coverage. The absorption properties of these dyes are detailed in Sections B-3 through B-6 of this report.

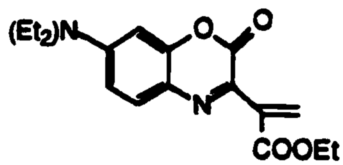
Finally, Spectra Science is in the process of arranging a strategic partnership with a commercial vendor to manufacture and market a full line of laser-protective eyewear. This line includes a number of NIR absorptive dyes developed during this program.

**Figure 1**
**Chemical Structure of Spectra Science Laser Protective Dyes Listed in Table 1**

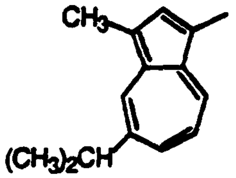
SS-1044*



SS-1100



SS-1045



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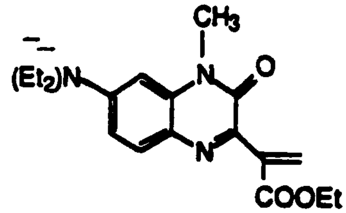

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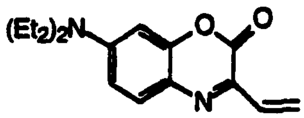

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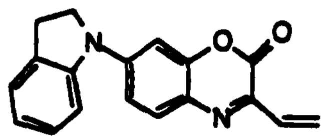

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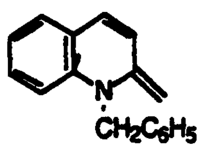

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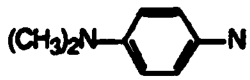

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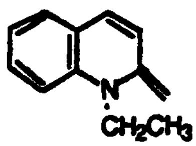

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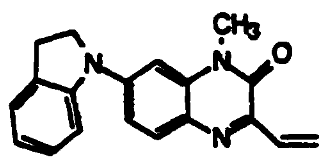

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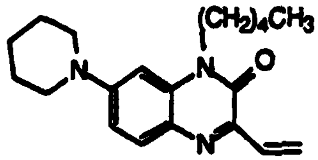

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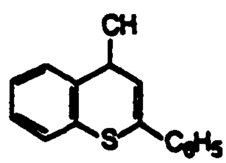

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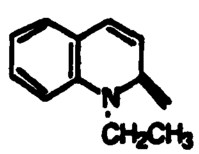

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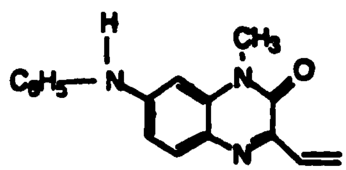
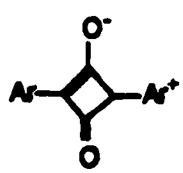


* where



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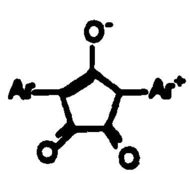

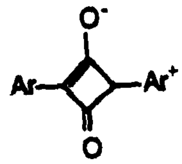

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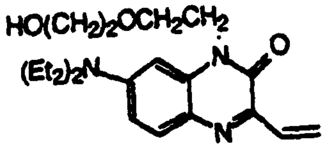

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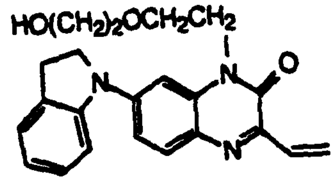

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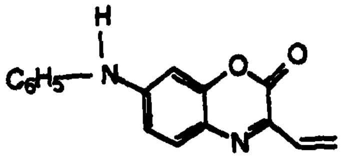

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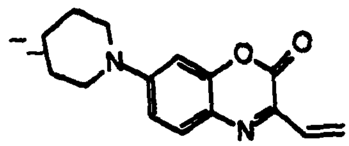

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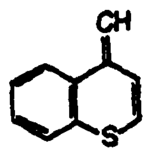

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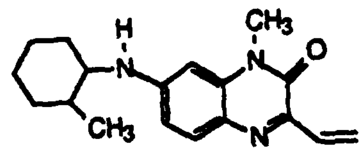

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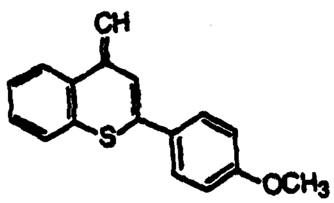

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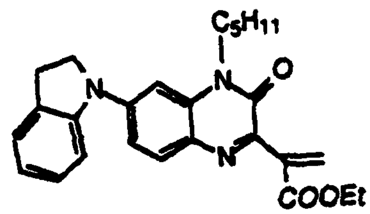

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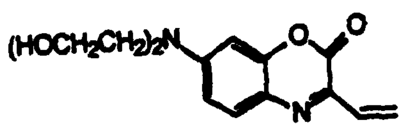

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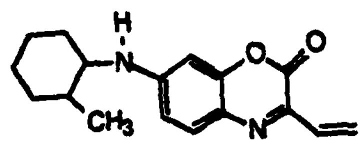

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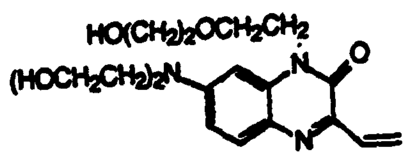

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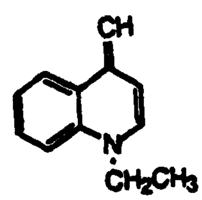

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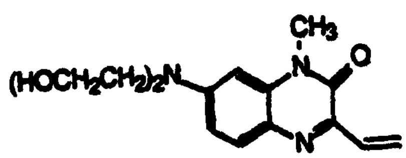

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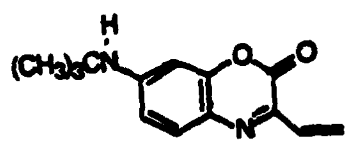

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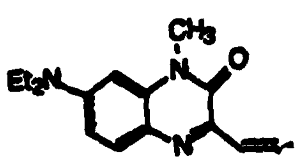

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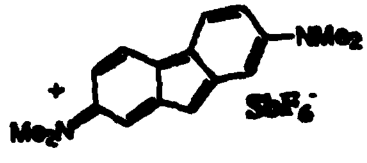
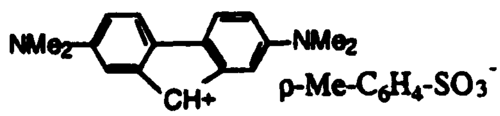


**Figure 1 (cont.)**
**Chemical Structure of Spectra Science Laser Protective Dyes Listed in Table 1**

SS-1140



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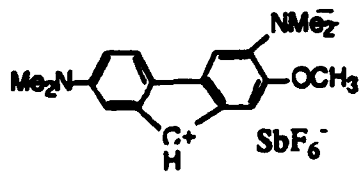

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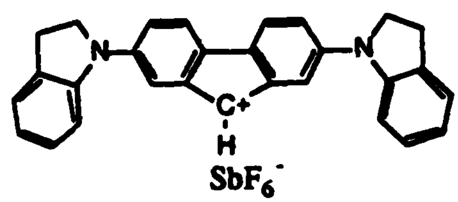

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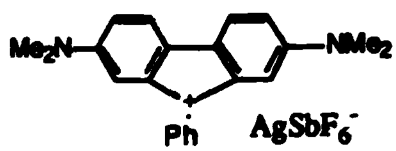

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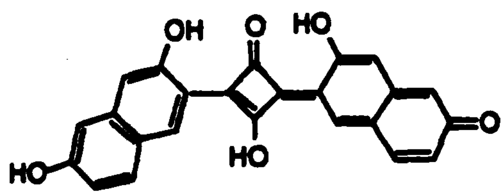

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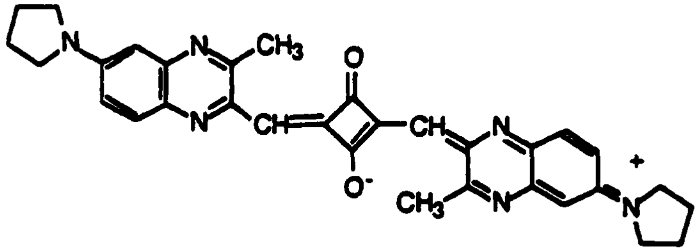

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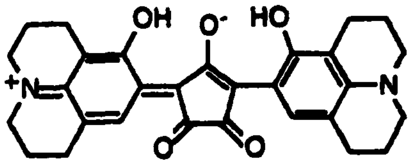

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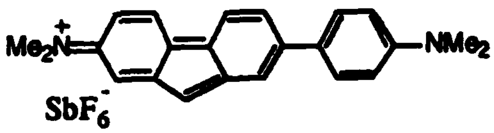

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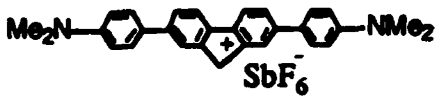

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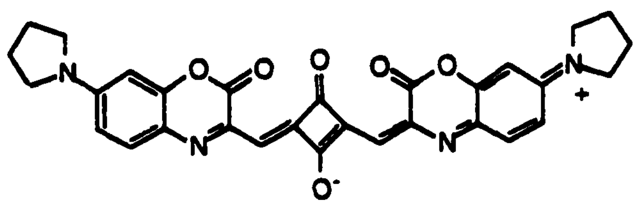

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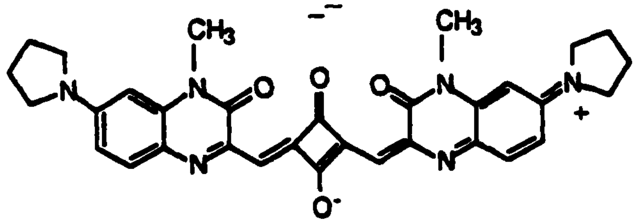

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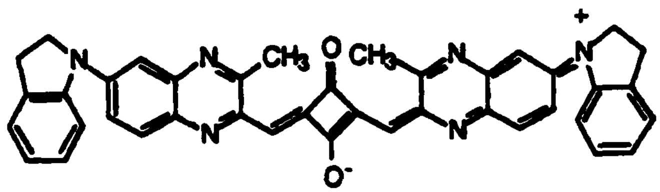

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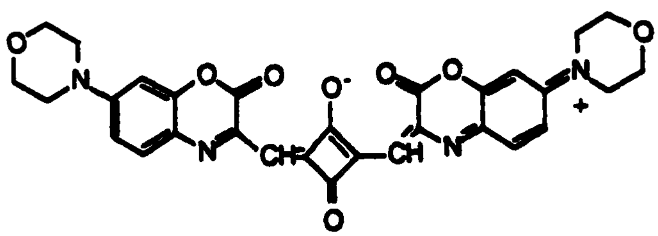

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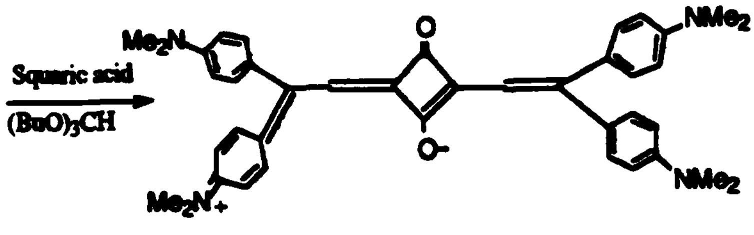


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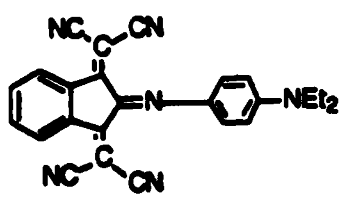
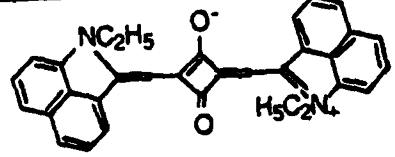


Figure 1 (cont.)
Chemical Structure of Spectra Science Laser Protective Dyes Listed in Table 1

SS-1166



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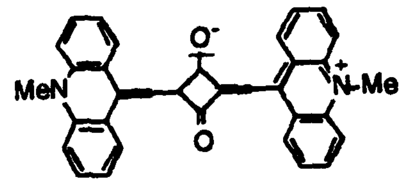

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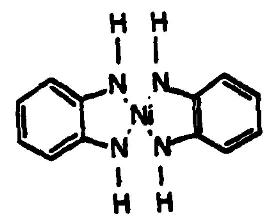
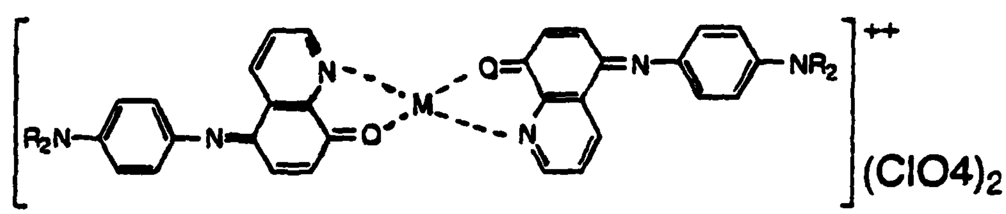
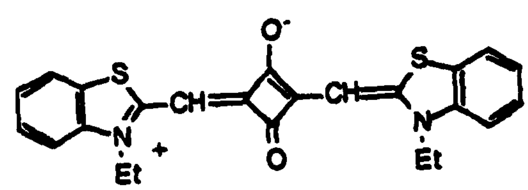


SS-1170 through SS-1173

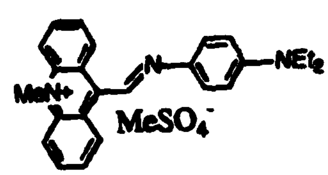


R = Me, Et
M = Cu, Ni

SS-1175



SS-1177



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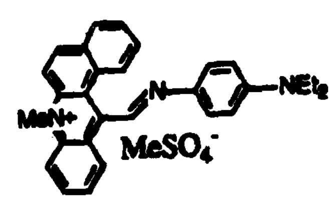
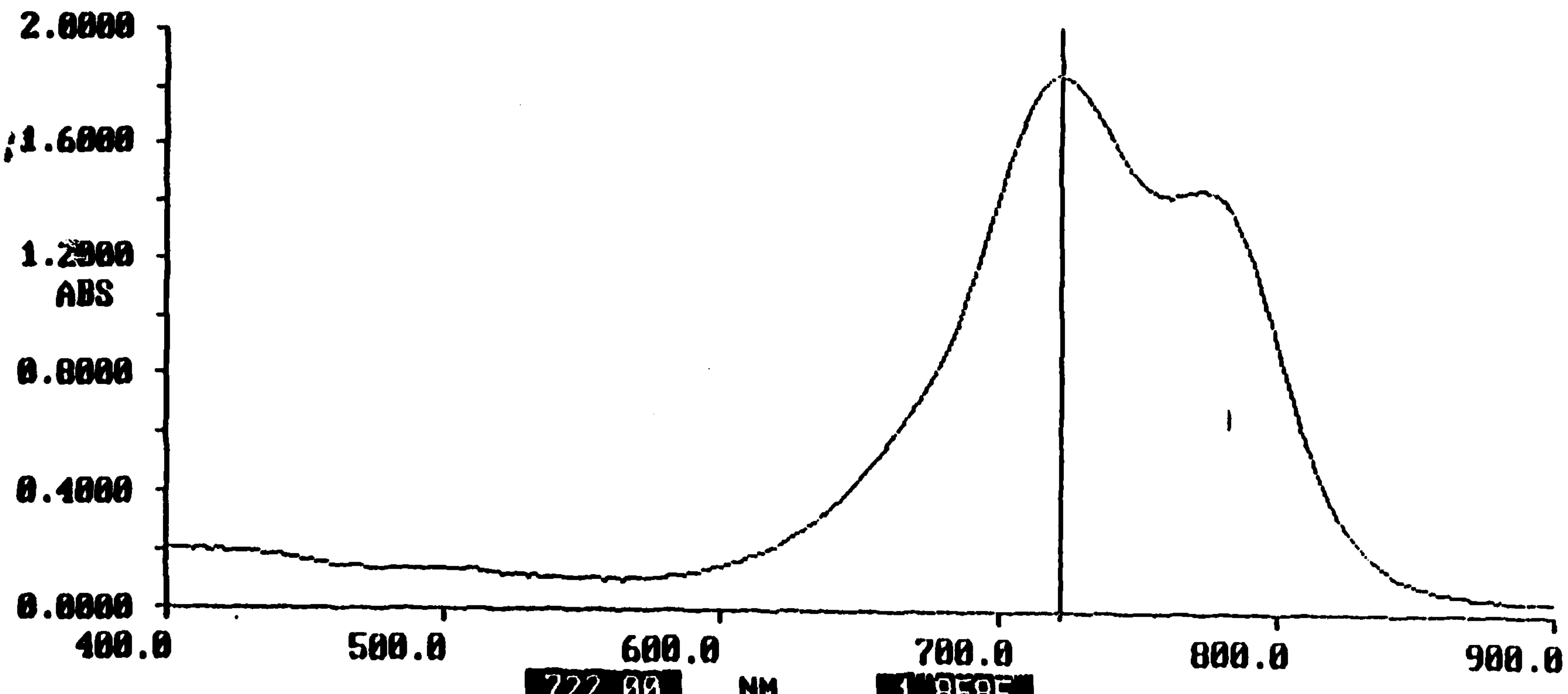


**SPECTRA SCIENCE**
From: Dr. Dennis P. Pacheco

**Figure 2**
Spectra Science Broad-Band Absorber for the Alexandrite Laser
(Spectrum is in Polycarbonate.)



**SPECTRA SCIENCE**
From: Dr. Dennis P. Pacheco

FIGURE 3

ABSORPTION SPECTRUM OF THE SQUARYLIUM DYE SF-1150 EXTRUDED INTO PETG FILM

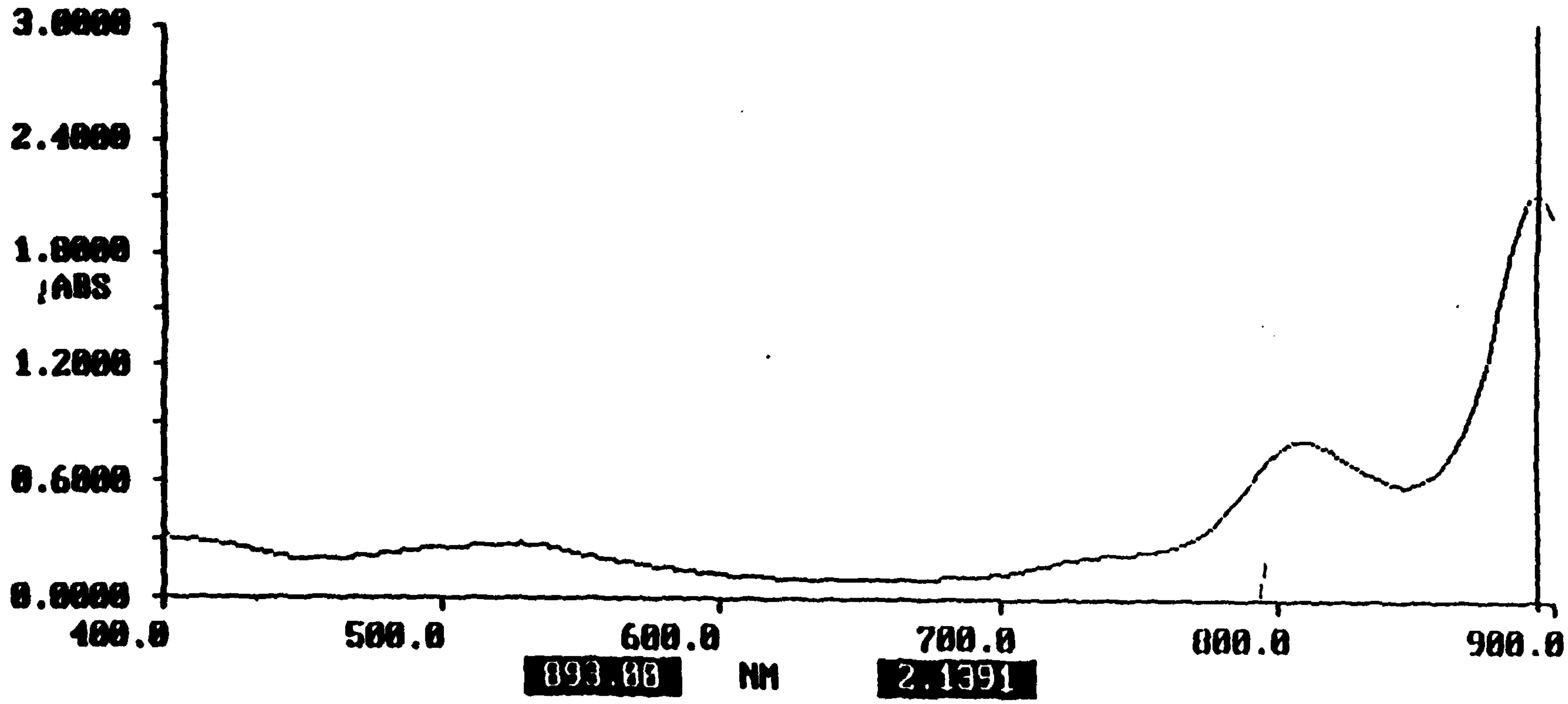


Figure 4 Absorption Spectrum of the Squarylium Dye SS-1151 in PC. The peak absorption is at approximately 860 nm.

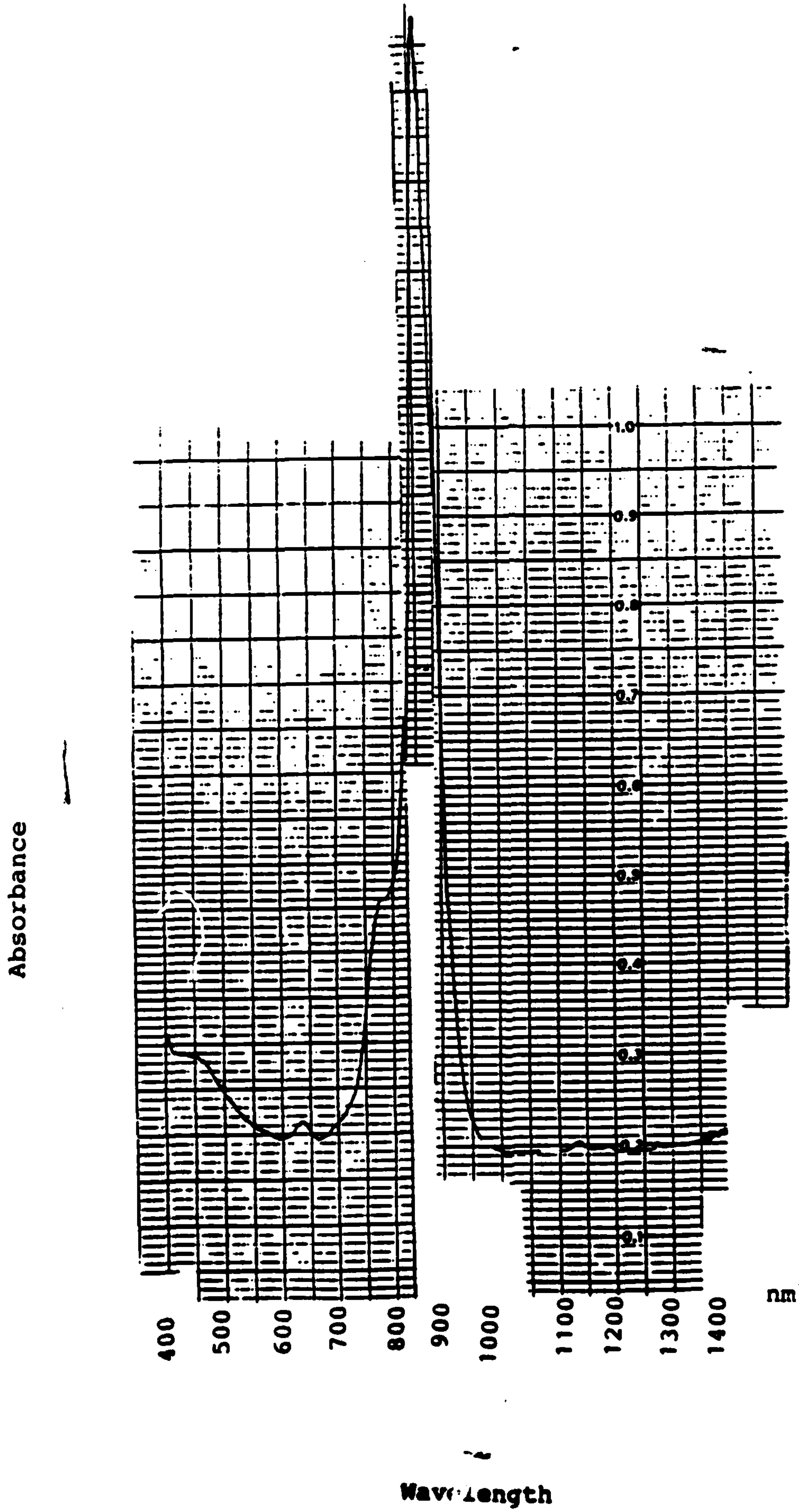


Figure. 5 Absorption Spectrum of the Squarylium Dye SS-1159 in PC. The peak absorption is at approximately 980 nm.